United States Environmental Protection Agency
Office of Enforcement and Compliance Assurance
Office of Criminal Enforcement, Forensics and Training

National Enforcement Investigations Center

NEIC

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NEIC CIVIL INVESTIGATION REPORT
3M Cordova
Cordova, Illinois 61242

Investigation Dates:
December 3-10, 2019

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General Appendices

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INVESTIGATION OVERVIEW

PROJECT OBJECTIVE

The U.S. Environmental Protection Agency (EPA) Office of Civil Enforcement (OCE) Water Enforcement Division (WED) and Waste and Chemical Enforcement Division (WCED) requested EPA's National Enforcement Investigations Center (NEIC) to conduct a multimedia compliance investigation of 3M Cordova, 22614 Route 84 North Cordova, Illinois 61242. The regulatory scope of the NEIC investigation was assessing 3M Cordova's compliance with the Clean Water Act (CWA) and the Resource Conservation and Recovery Act (RCRA). The focus of the investigation was to conduct on-site process evaluations with emphasis on waste management and per- and poly-fluoroalkyl substances (PFAS) wastewater generation, management, treatment, and effluent discharge. PFAS are a group of man-made chemicals that include perfluorooctanoic acid (PFOA), perfluorooctane sulfonic acid (PFOS), and many other fluorine-containing chemicals.

The project team members are listed in **Table 1**.

Table 1. PROJECT TEAM MEMBERS

Team Member	Organization	Project Role
Daren Vanlerberghe	NEIC	Project manager (PM)
David Parker	NEIC	Field team member
Trent Rainey	NEIC	Field team member
Linda TeKrony	NEIC	Field team member
Stephanie Volz	NEIC	Principal analytical chemist (PAC), laboratory team lead
Dan Hurlbut	NEIC	Laboratory team member
Adrian Krawczeniuk	NEIC	Laboratory team member and laboratory quality assurance representative

FACILITY CONTACT INFORMATION

Table 2 lists the primary facility contacts. Credentials were presented to David Andrews and Rich Stutzki on December 3, 2019.

Table 2. FACILITY CONTACT INFORMATION

Name, Title	Phone No.	Email Address
David Andrews, Plant Manager	309-654-8056	djandrews1@mmm.com
Rich Stutzki, Environmental Health, Safety, and Regulatory Manager	309-654-8070	rstutzki@mmm.com

FACILITY OVERVIEW

According to the EPA Envirofacts database, this facility has the following North American Industry Classification System (NAICS) codes (**Table 3**):

Table 3. APPLICABLE NAICS CODES	
NAICS Code	**Description**
325211	Plastics and resin manufacturing
325520	Adhesive manufacturing
325130	Synthetic dye and pigment manufacturing
325131	Inorganic dye and pigment manufacturing
325998	All other miscellaneous chemical product and preparation manufacturing

3M Cordova operates a specialty chemical and adhesive manufacturing facility (EPA Registry Identification No. 110013886875). According to a site overview provided by 3M Cordova during the inspection (**Appendix A**), the facility site is located on approximately 750 acres along the Mississippi River, of which 125 are developed. Production at the site started in 1970, and there are currently 470 employees at the site.

Clean Water Act

3M Cordova is authorized to discharge treated wastewater and stormwater to the Mississippi River under National Pollutant Discharge Elimination System (NPDES) permit No. IL0003140 (**Appendix CWA A**). The permit was issued by Illinois EPA and became effective on January 1, 2013. The permit had an expiration date of December 31, 2017; however, the permit has been administratively extended and remains in effect. The permit authorizes 3M Cordova to discharge treated wastewater, noncontact cooling water (NCCW), and stormwater runoff through five outfalls (001, A01, 002, 003, and 004). The permit contains effluent monitoring requirements at outfall 001 for 14 PFAS.

Resource Conservation and Recovery Act

3M Cordova is a RCRA large quantity generator (LQG) of hazardous waste (EPA ID No. ILD054236443). At one time, 3M Cordova was a permitted treatment storage and disposal facility (TSDF) operating an incinerator, surface impoundment, tanks, and container storage areas under a hazardous waste permit. The last permitted unit was closed in June 1997. NEIC conducted a compliance inspection of the facility in March 2018 focusing on RCRA air emission requirements. At the time of the March 2018 NEIC on-site inspection, 3M Cordova was operating four less-than-90-day hazardous waste accumulation areas (90-day areas).

FACILITY OPERATIONS SUMMARY

Production at 3M Cordova includes specialty fluorochemical finished goods used in electronics and fire protection industries and semi-finished fluorochemicals used to manufacture carpet and apparel products, battery electrolytes, and specialty monomers and surfactants.

Production at 3M Cordova also includes solvent-less adhesives, acrylate monomers, various adhesives and low-adhesion backsizes, and medical products.

Appendix B contains a description of the major manufacturing process units and utilities at 3M Cordova, focusing on waste and wastewater stream generation. **Appendix B** also includes a description of the process wellfield used for 3M Cordova's process and cooling source water and a description of 3M Cordova's on-site wastewater treatment plant (WWTP). The description is based on process flow diagrams and verbal descriptions provided by 3M Cordova staff during the inspection, as well as discussions and observations made during the inspection, and contains company-claimed confidential business information.

FIELD ACTIVITIES SUMMARY

Daren Vanlerberghe, David Parker, and Trent Rainey from NEIC conducted a CWA on-site inspection at 3M Cordova from December 3-10, 2019. Linda TeKrony from NEIC conducted a RCRA on-site inspection at 3M Cordova from December 3-6, 2019. Ray Cullen from EPA Region 5 and Sean Ireland from EPA OCE also participated in portions of the inspection. **Appendix C** contains sign-in sheets documenting participants for the opening meeting, RCRA inspection closing meeting, and CWA/overall inspection closing meeting.

NEIC inspected process areas, waste and wastewater generation sources, waste and wastewater management and treatment areas, and wastewater discharge and self-monitoring locations. Specific CWA and RCRA areas inspected by NEIC are discussed below. NEIC collected samples as part of the inspection, and sampling activities are described in detail later in this report. NEIC also interviewed 3M Cordova personnel, reviewed documents, and took photographs as part of the inspection. NEIC inspection photographs are included in **Appendix CWA B** and **Appendix RCRA C**.

Clean Water Act

NEIC inspected process areas, wastewater generation sources, wastewater management and treatment areas, stormwater management areas, and wastewater discharge and self-monitoring locations. NEIC reviewed facility records, including sewer maps and diagrams, standard operating procedures, calibration records, sampling records, monitoring reports, permits, and permit applications.

Stormwater Management

While a full facility stormwater inspection was not part of the scope of the NEIC investigation, NEIC did discuss 3M Cordova's stormwater management, reviewed stormwater drainage maps and certain plans and records, inspected selected stormwater management areas, inspected

stormwater outfalls, and sampled impounded wastewater/stormwater at selected stormwater outfall locations (no discharges from the facility stormwater outfalls occurred during the inspection to NEIC's knowledge).

3M Cordova's NPDES permit includes three listed and authorized stormwater outfalls (002, 003, and 004), all listed in the permit as "Intermittent Discharge." Stormwater outfalls are located along the west side of the facility near the Mississippi River, with outfall numbering ascending from north to south. 3M Cordova manages stormwater runoff collected for discharge through outfalls 003 and 004 by storing it behind a normally closed sluice gate prior to its discharge. At outfall 002, there is no discharge gate structure, and stormwater runoff flows freely prior to discharging through the outfall.

During the inspection, NEIC discovered that 3M Cordova also refers to outfalls 002, 003, and 004 as outfalls A, C, and D, respectively (e.g., in the storm water pollution prevention plan [SWPPP] and NPDES permit renewal applications), and also as outlets A, C, and D, respectively (e.g., on site drainage maps). During review of maps and records, NEIC also discovered that 3M Cordova has an outfall B (or outlet B) discussed in the SWPPP and included on site drainage maps and an outlet A-1 included on site drainage maps (not included in the SWPPP), both located on the west side of the facility along the Mississippi River. (NEIC does not have electronic copies of site drainage maps due to the size of the maps, but does have hard copies available.) Neither outfall B nor outlet A-1 is included in 3M Cordova's current NPDES permit.

3M Cordova's 2007 NPDES permit renewal application (application for the current effective permit) and 2017 NPDES permit renewal application (application for the permit renewal yet to occur) both include only outfalls 002 (A), 003 (C), and 004 (D) listed on EPA Form 2F (**Appendices CWA C** and **CWA D**, pages 51 and 45, respectively). However, both permit renewal applications include a narrative reference in Attachment 2F-IVC to outfall B (**Appendices CWA C** and **CWA D**, pages 66 and 58, respectively): "The structural controls found in the drainage area for Outfall B include indoor storage of hazardous waste and raw materials and a gated structure that is closed to contain spills or contaminated storm water to allow transfer to the wastewater treatment plant, if necessary." Both permit renewal applications also state, "The gate to Outfall B is never opened." During the inspection, Keith Schmuck, 3M Cordova quality manager, stated that the gate at outfall B stays closed, and impounded water behind the gate either percolates or evaporates.

Outlet A-1 is not listed or mentioned in 3M Cordova's SWPPP or in the permit renewal applications. On site drainage maps, outlet A-1 is shown in the extreme northwest area of the facility along the Mississippi River. During the inspection on December 5, 2019, NEIC and 3M Cordova representatives attempted to locate outlet A-1; no discrete outlet or conveyance was

found or observed. The location of outlet A-1 on the site drainage maps is a wooded area, with no observed industrial activity at the time of the inspection.

Resource Conservation and Recovery Act

NEIC collected information regarding environmental monitoring conducted by the facility for PFAS. As part of collecting information regarding environmental monitoring conducted by the facility, NEIC reviewed: April 2014 *Environmental Assessment for Perfluorochemicals (PFCs) Summary Report*, 2018 *Annual PFAS Monitoring Report for the 3M Cordova, IL Facility*, and *RCRA Facility Assessment*. NEIC inspected the following 90-day areas: loading station 16, building 18, building 30, building 2, building 3 pit (or "silo"), and loading station 83.

3M Cordova operated 12 sludge field zones from 1975 through 1999. These sludge field zones were permitted by Illinois EPA, originally in 1975, with a supplemental permit granted in 1976. The facility renewed the permit continually from 1976 until sludge incorporation was discontinued at the end of December 1999. The zones were used during the following time frames:

- Zones 1-9: 1975-1999
- Zone 10: 1993, one time in 1998
- Zone 11: Never used
- Zone 12: 1994-1999

The sludge field zones are areas where sludge from the on-site wastewater treatment plant was amended into the soil. The sludge was placed on the land and then tilled into the soil with a disc. Spreading rates averaged 8,000 gallons per acre per year. Sludge was not applied in the winter when the ground was frozen, during periods of rainfall, or on ground with standing water.

The four sludge field zones located west of the highway (zones 5, 6, 7, and 9) were planted with agricultural crops during the sludge amendment time frame. These same four sludge field zones were remediated by planting native prairie plant species in 2003 when the growing of crops was discontinued.

Site Characterization

3M Cordova conducted a site characterization from 2006 through 2013 (**Appendix RCRA A**) that focused on five compounds. The compounds are listed in the characterization report as: perfluorooctane sulfonate (PFOS), perfluorooctanoic acid (PFOA), perfluorobutanoic acid (PFBA), perfluorohexane sulfonate (PFHS), and perfluorobutane sulfonate (PFBS). Note that this inspection report uses different compound designations and abbreviations for two chemicals, perfluorooctane sulfonate acid (PFOS) and perfluorohexane sulfonate (PFHxS).

During this same time frame, 3M Cordova discontinued the manufacture of perfluoro-octanyl compounds. In 2014, Illinois EPA designated the groundwater under the facility a groundwater management zone. As required by the groundwater designation, 3M Cordova conducts semi-annual sampling of facility monitoring wells, and annual sampling of residential wells located adjacent to the facility's eastern boundary. The facility monitoring wells are located throughout the sludge field zones.

Production wells for facility process water are located within the groundwater management zone. These wells capture the groundwater before it can leave the facility area. Even though the Mississippi River is located to the west of the facility, the groundwater flows to the east, away from the river. 3M Cordova groundwater elevation measurements confirm the flow of groundwater away from the Mississippi River, which indicates "effective capture of impacted groundwater at the Site." The process water is used throughout the facility. Used process water is treated in the WWTP before being discharged to the Mississippi River. NCCW water is used throughout the plant and does not undergo any treatment before being mixed with the treated wastewater and discharged to the Mississippi River.

Based on the trends of the data collected between 2014 and the present, 3M Cordova believes the concentrations of the five site characterization compounds are declining in the groundwater. 3M Cordova's most recent annual PFAS monitoring report (**Appendix RCRA B**) contains the data generated throughout the study of the groundwater.

3M Cordova conducted soil sampling and analysis on three different occasions: March 2011, April/May 2012, and April 2013. Remediation objectives (contained in Appendix C to the site characterization [**Appendix RCRA A**]) set by Illinois EPA have baseline objectives for each of the five site characterization compounds. According to the site characterization (**Appendix RCRA A**, page 22), the objectives for PFOS were not met for residential ingestion and soil-to-groundwater ingestion. The data used to make this conclusion were submitted to Illinois EPA in February 2014 in a document titled *2013 Assessment Activities Summary Report*. NEIC did not request a copy of this document during the on-site inspection. No additional sampling or analysis on soils has occurred since this 2013 assessment.

NEIC requested information regarding the RCRA facility assessment conducted at 3M Cordova. The assessment was completed in 1998 but did not address or consider constituents beyond the Appendix IX RCRA constituents. Solid waste management units identified during the assessment were the sludge field zones and the wastewater treatment plant area.

Sampling and/or Measurement Activities

All environmental measurement activities were performed in accordance with the NEIC quality system. All field sampling activities described in this report are within the scope of NEIC's ISO/IEC 17025 accreditation issued by the ANSI National Accreditation Board (certificate No. AT-1646). Samples collected by 3M staff are not covered under NEIC's accreditation.

Table 4 summarizes field sampling activities. A copy of the electronic transfer of custody record is provided in **Appendix D**. NEIC CWA sample descriptions, activities, observations, and chain of custody notes and records are documented in the project file. Specifically, the practices NEIC used during the CWA sampling to help prevent interferences and potential cross-contamination of PFAS samples are also documented in the project file, including selection and use of sampling equipment, personal protective equipment (PPE), clothing, and hygiene practices. Pertinent photographs from the sampling activities are provided in **Appendix CWA B**.

Table 4. FIELD SAMPLING PERFORMED

Location Identifier (Station/Sample No.)	Date and Time	Method, and/ or Procedure, and Equipment	Sampler/ Measurer Names
Station S01 – NEIC Sample No. VP1364-01 Trip blank 1 (TB-1)	11/20/2019 at 09:30	Trip blanks and field spikes **Sample containers**: 1-liter high density polyethylene (HDPE) plastic container (without Teflon™ -lined lids); nitric acid preservative	Prepared by Dan Hurlbut, NEIC analyst, in advance of the inspection and provided to Daren Vanlerberghe, NEIC project manager, to bring in the field
Station S01 – NEIC Sample No. VP1364-02 Trip blank 2 (TB-2)	11/20/2019 at 09:30		
Station S01 – NEIC Sample No. VP1364-03 Trip blank 3 (TB-3)	11/20/2019 at 09:30		
Station S01 – NEIC Sample No. VP1364-04 Low field spike	11/20/2019 at 09:30		
Station S01 – NEIC Sample No. VP1364-05 Medium field spike	11/20/2019 at 09:30		
Station S01 – NEIC Sample No. VP1364-06 High field spike	11/20/2019 at 09:30		
Station S02 – Production wellfield Building 10 Sample No. VP1364-07	12/6/2019 at 08:57	**Method:** ASTM D5013-18[1] and ASTM D5358-93[2] (used as guidance)	Trent Rainey Daren Vanlerberghe

Table 4. FIELD SAMPLING PERFORMED			
Location Identifier (Station/Sample No.)	**Date and Time**	**Method, and/ or Procedure, and Equipment**	**Sampler/ Measurer Names**
Station S03 – Stormwater outfall 004 (sample collected from impounded wastewater/stormwater upstream of the closed sluice gate, no discharge occuring at time of sample collection) Sample No. VP1364-08	12/6/2019 at 09:48	**NEIC procedure:** *Water and Wastewater Sampling,* NEICPROC/00-051 **Equipment:** Grab sample using direct fill into sample container or with sample container affixed to telescoping rod **Sample containers**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Trent Rainey Daren Vanlerberghe
Station S04 – Field equipment rinse blank Sample No. VP1364-09	12/6/2019 at 10:00		
Station S05 – Stormwater outlet B (sample collected from impounded wastewater/stormwater upstream of the closed sluice gate, no discharge occuring at time of sample collection) Sample No. VP1364-10	12/6/2019 at 10:33		
Station S06 – Field blank Sample No. VP1364-11	12/6/2019 at 10:40		
Station S07 – Outfall 001 (DTR) Sample No. VP1364-12	12/6/2019 at 11:08		
Station S07 – Outfall 001 (DTR) Sample No. VP1364-13	12/6/2019 at 11:14		
Station S07 – Outfall 001 (DTR) Sample No. VP1364-14	12/6/2019 at 11:20		
Station S08 – Outfall A01 (Pond #3) Sample No. VP1364-15	12/6/2019 at 12:57		
Station S09 – Lift station B63 Sample No. VP1364-16	12/6/2019 at 13:22		
Station S10 – Building 30 and thermal oxidizer (TO) discharge Sample No. VP1364-17	12/6/2019 at 13:57		
Station S11 – Building 20 discharge Sample No. VP1364-18	12/6/2019 at 14:09		

[1] ASTM D5013-18: Standard Practices for Sampling Wastes from Pipes and Other Point Discharges
[2] ASTM D5358-93: Standard Practice for Sampling with a Dipper or Pond Sampler

LABORATORY ACTIVITIES SUMMARY

Laboratory Analytical Activities

Table 5 summarizes the laboratory team's analysis of the samples.

Table 5. ANALYTICAL OBJECTIVE, TECHNIQUE, METHOD; ANALYST; AND DATE PERFORMED

Analytical Objective, Technique, and Method and/or NEIC Procedure[1]	NEIC Analyst	Samples Analyzed by Method (Station No.)	Dates Performed
PFAS water analysis #1: • Improved Method for Extraction and Analysis of Perfluorinated Compounds (PFCs) from Surface Waters and Well Water by Ultra-High Performance Liquid Chromatography (UPLC)-Tandem Mass Spectrometry (MS/MS). SOP ID: D-EMMDPHCB043-SOP-03, August 2016. • Method for Extraction and Analysis of Perfluoroethercarboxylic acids (PFECAs) from Surface Water, Well Water and Waste Water by Ultra-High Performance Liquid Chromatography (UPLC)-Tandem Mass Spectrometry (MS/MS). SOP ID:D-EMMD-PHCB-062-SOP-01, July 2017. • Samples analyzed by liquid chromatography/mass spectrometry (LC/MS) using a triple-quadrupole (QQQ) mass spectrometer for detection of targeted PFAS	Dan Hurlbut	VP1364-01—VP1364-18	Sample loading on cartridge: December 17-20, 2019 Sample elution: January 21, 2020 Analysis: January 22-April 2, 2020
PFAS water analysis #2: • Same methodologies and extracts as analysis #1 • Samples analyzed by LC/MS using a quadrupole time of flight (QTOF) mass spectrometer for detection of non-target PFAS	Stephanie Volz	VP1364-01—VP1364-18	Analysis: January 22-March 10, 2020

[1] Data quality summaries, including uncertainty measurements, for all laboratory measurements are maintained in the project file.

All environmental measurement activities were performed in accordance with the NEIC quality system. All laboratory analyses described in this report are within the scope of NEIC's ISO/IEC 17025 accreditation issued by the ANSI National Accreditation Board (certificate No. AT-1646).

INVESTIGATION ANALYTICAL RESULTS

Table 6 shows the station number, the sampling station location, the NEIC tag number, and the laboratory description for the samples analyzed. NEIC laboratory personnel prepared three blank samples (VP1364-01, VP1364-02, and VP1364-03) and three field spikes (VP1364-04, VP1364-05, and VP1364-06) and provided them to NEIC field inspectors prior to sample collection. These six samples travelled to and from the field site and were shipped with the other samples. The PFAS concentrations in the field spikes were 25 nanograms per liter (ng/L) (VP1364-04), 50 ng/L (VP1364-05), and 100 ng/L (VP1364-06).

Table 6. STATION NUMBERS, STATION LOCATIONS, NEIC TAG NUMBERS, AND LABORATORY DESCRIPTION

Station No.	Station Location	NEIC Tag No.	Laboratory Description
S01/NEIC	NEIC (into field and return)	VP1364-01	Colorless, clear, non-viscous liquid
S01/NEIC	NEIC (into field and return)	VP1364-02	Colorless, clear, non-viscous liquid
S01/NEIC	NEIC (into field and return)	VP1364-03	Colorless, clear, non-viscous liquid
S01/NEIC	NEIC (into field and return)	VP1364-04	Colorless, clear, non-viscous liquid
S01/NEIC	NEIC (into field and return)	VP1364-05	Colorless, clear, non-viscous liquid
S01/NEIC	NEIC (into field and return)	VP1364-06	Colorless, clear, non-viscous liquid
S02/Production wellfield building 10	Production wellfield building 10	VP1364-07	Colorless, clear, non-viscous liquid
S03/Stormwater outfall 004	Collection area behind/upstream of outfall 004 sluice gate	VP1364-08	Light-brown non-viscous liquid with a visible amount of particulate
S04/Field equipment rinse blank	Near stormwater outfall 004	VP1364-09	Colorless, clear, non-viscous liquid
S05/Stormwater outlet B	Collection area behind/upstream of outlet B sluice gate	VP1364-10	Light-brown non-viscous liquid with a visible amount of particulate
S06/Field blank	Near stormwater outlet B	VP1364-11	Colorless, clear, non-viscous liquid
S07/Outfall 001 (DTR)	Discharge channel downstream of 3M DTR sample shack	VP1364-12	Colorless, clear, non-viscous liquid
S07/Outfall 001 (DTR)	Discharge channel downstream of 3M DTR sample shack	VP1364-13	Colorless, clear, non-viscous liquid with a very small amount of light-brown particulate post filtering
S07/Outfall 001 (DTR)	Discharge channel downstream of 3M DTR sample shack	VP1364-14	Colorless, clear, non-viscous liquid with a very small amount of light-brown particulate post filtering
S08/Outfall A01 (Pond #3)	Discharge stream directly downstream of the effluent flow monitoring V-notch weir	VP1364-15	Light-brown non-viscous liquid with a visible amount of particulate
S09/Lift station B63	Wet well in building of lift station B63	VP1364-16	Colorless, clear, non-viscous liquid
S10/Building 30 and thermal oxidizer (TO) discharge	Discharge pipe from building 30 and TO	VP1364-17	Colorless, clear, non-viscous liquid with a very small amount of light-yellow sediment post filtering
S11/Building 20 discharge	Discharge pipe from building 20	VP1364-18	Colorless, clear, non-viscous liquid

In general, the samples shown in **Table 6** were clear, colorless, non-viscous liquids, some with particulates. The extraction and analysis methods shown in **Table 5** were developed and validated by EPA analysts at the Office of Research and Development (ORD) National Exposure Research Laboratory, for PFAS determinations in water matrices that were similar to the

samples shown in Table 6. Each sample was passed through a glass fiber filter in order to remove any particulate present, prior to extraction. Sample VP1364-15 (outfall A01 (pond #3)) required five filters to remove the particulate material that was present. Samples VP1364-08, VP1364-10, VP1364-13, VP1364-14, and VP1364-17 required only a single filter to remove the particulate material that was present. No particulate material was observed in the remaining 12 samples. Additional information on the laboratory activities, including sample preparation, filtration, extraction, spike recoveries and reporting limits (R.L.), can be found in **Appendix E**. Samples VP1364-01—VP1364-18 are CWA samples that were extracted and analyzed for PFAS using methodologies and LC/MS techniques listed in **Table 5**.

Diluted sample extracts were analyzed using two different mass spectrometers. The triple-quadrupole (QQQ) mass spectrometer utilized certified reference materials to identify and quantify the 36 compounds listed in **Table 7**, using analyte-specific detection parameters. In addition, the QQQ analysis included analyzing the water samples directly. The quadrupole time-of-flight (QTOF) mass spectrometer was used to determine the presence of an additional 34 PFAS listed in **Table 11** in the extracts only. These determinations were made by comparing observed accurate mass values to the exact mass values for these perfluorinated compounds, and where reference materials were available, by comparing retention time and fragmentation patterns. In summary, the QQQ provided quantitative results, as this system was specifically set up to analyze the extracts for those compounds shown in **Table 7**. Results from the QTOF analysis are qualitative identifications based on accurate mass data that were collected for all responses within a 50-to-1000 mass unit range. Therefore, results from the two instruments will be different based on the functionality of each mass spectrometer.

LC/MS QQQ ANALYSIS

Target Analytes

Table 7 shows the 36 target PFAS for the LC/MS QQQ analysis. In addition to the diluted extracts, a set of diluted filtered subsample aliquots ("Diluted #1") was prepared and analyzed for these PFAS. When the Diluted #1 analysis set was processed, six of the target analytes had concentration values that exceeded the calibration curve. Therefore, a second set of diluted filtered subsample aliquots ("Diluted #2") was prepared and analyzed using calibration standards with higher analyte concentrations.

Table 7. TARGET ANALYTES, ABBREVIATIONS AND MOLECULAR FORMULAS

Compound Class	Analyte Name	Abbreviation	Formula
Carboxylic acids	Perfluorobutyric acid[1]	PFBA	$C_4HF_7O_2$

Table 7. TARGET ANALYTES, ABBREVIATIONS AND MOLECULAR FORMULAS			
Compound Class	**Analyte Name**	**Abbreviation**	**Formula**
	Perfluorodecanoic acid[1]	PFDA	$C_{10}HF_{19}O_2$
	Perfluorododecanoic acid[1]	PFDoA	$C_{12}HF_{23}O_2$
	Perfluoroheptanoic acid[1]	PFHpA	$C_7HF_{13}O_2$
	Perfluorohexadecanoic acid	PFHxDA	$C_{16}HF_{31}O_2$
	Perfluorohexanoic acid[1]	PFHxA	$C_6HF_{11}O_2$
	Perfluorononanoic acid[1]	PFNA	$C_9HF_{17}O_2$
	Perfluorooctadecanoic acid	PFODA	$C_{18}HF_{35}O_2$
	Perfluorooctanoic acid[1]	PFOA	$C_8HF_{15}O_2$
	Perfluoropentanoic acid[1]	PFPeA	$C_5HF_9O_2$
	Perfluorotetradecanoic acid	PFTeA	$C_{14}HF_{27}O_2$
	Perfluorotridecanoic acid[1]	PFTrA	$C_{13}HF_{25}O_2$
	Perfluoroundecanoic acid[1]	PFUnA	$C_{11}HF_{21}O_2$
Ether carboxylic acids	Undecafluoro-2-methyl-3-oxahexanoic acid	GenX	$C_6HF_{11}O_3$
	Butanoic acid, 2,2,3,3,4,4-hexafluoro-4-(trifluoromethoxy)-	PFECA A	$C_5HF_9O_3$
	Acetic Acid, 2,2-difluoro-2-[1,1,2,2-tetrafluoro-2-(trifluoromethoxy)ethoxy]-	PFECA B	$C_5HF_9O_4$
	Butanoic acid, 2,2,3,3,4,4-hexafluoro-4-[1,2,2,2-tetrafluoro-1-(trifluoromethyl)ethoxy]-	PFECA G	$C_7HF_{13}O_3$
Sulfonamides	N-ethylperfluoro-1-octanesulfonamide	N-EtFOSA	$C_{10}H_6F_{17}NO_2S$
	N-methylperluoro-1-octanesulfonamide	N-MeFOSA	$C_9H_4F_{17}NO_2S$
	Perfluorooctanesulfonamide[1]	FOSA	$C_8H_2F_{17}NO_2S$
Sulfonamidoacetic acids	N-ethyl-N-((heptadecafluorooctyl)sulfonyl)glycine	N-EtFOSAA	$C_{12}H_8F_{17}NO_4S$
	N-(Heptadecafluorooctylsulfonyl)-N-methylglycine	N-MeFOSAA	$C_{11}H_6F_{17}NO_4S$
Sulfonic acids	Perfluorobutanesulfonic acid[1]	PFBS	$C_4F_9O_3SH$
	Perfluorodecanesulfonic acid	PFDS	$C_{10}F_{21}O_3SH$
	Perfluorododecylsulfonic acid[2]	PFDoS	$C_{12}F_{25}O_3SH$
	Perfluoroheptanesulfonic acid	PFHpS	$C_7F_{15}O_3SH$
	Perfluorohexanesulfonic acid[1,3]	(B,L) PFHxS	$C_6F_{13}O_3SH$
	Perfluorononanesulfonic acid	PFNS	$C_9F_{19}O_3SH$
	Perfluorooctanesulfonic acid[1,3]	(B,L) PFOS	$C_8F_{17}O_3SH$
	Perfluoropentansulfonic acid	PFPeS	$C_5F_{11}O_3SH$

Table 7. TARGET ANALYTES, ABBREVIATIONS AND MOLECULAR FORMULAS			
Compound Class	**Analyte Name**	**Abbreviation**	**Formula**
Telomer sulfonic acids	4:2 Fluorotelomer sulfonic acid	4:2 FTS	$C_6H_5F_9O_3S$
	6:2 Fluorotelomer sulfonic acid	6:2 FTS	$C_8H_5F_{13}O_3S$
	8:2 Fluorotelomer sulfonic acid	8:2 FTS	$C_{10}H_5F_{17}O_3S$
	10:2 Fluorotelomer sulfonic acid	10:2 FTS	$C_{12}H_5F_{21}O_3S$
Sulfonamido ethanols	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	EtFOSE	$C_{12}H_{10}F_{17}NO_3S$
	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	MeFOSE	$C_{11}H_8F_{17}NO_3S$

[1] Analytes are listed in the facility's NPDES permit for outfall 001
[2] Analyte present in second source mix only and was used for identification and estimation purposes, if present in a sample.
[3] Analyte in stock mixture is comprised of branched (B) and linear (L) isomers.

Results

The results in **Tables 8, 9**, and **10** are reported as either the observed PFAS concentrations, not observed, or identified at a concentration less than the reporting limit, with the estimated measurement uncertainty (EMU) for each target analyte for samples VP1364-07–VP1364-18. Applicable results from all three analysis sets were used in generating these tables.

The "total" designation for PFHxS and PFOS indicates analyte concentrations that include linear and branched isomers. In addition, five target analytes were not detected in any field samples, and these results were omitted from Tables 8, 9, and 10:

- 1 carboxylic acid: PFTrA (listed in the facility's NPDES permit for outfall 001)
- 2 sulfonamides: N-EtFOSA, N-MeFOSA
- 1 sulfonic acid: PFDoS
- 1 sulfonamidoacetic acid: N-MeFOSAA

Three other target analytes, PFTeA, PFHxDA and PFODA, were not observed in the samples as well as fortified laboratory samples, and were excluded from Tables 8, 9, and 10.

Table 8. OBSERVED PFAS CONCENTRATIONS, R.L. AND EMU VALUES FOR SAMPLES VP1364-07, VP1364-08, AND VP1364-10							
Analyte	*Analyte Concentration (ng/L)*						EMU (±%)
	VP1364-07		VP1364-08		VP1364-10		
	Sample	*R.L.*	Sample	*R.L.*	Sample	*R.L.*	
PFBA	**10,800**	*1,000*	**36,800**	*1,000*	**6,490**	*1,000*	15
PFDA	**193**	*3.78*	**17.6**	*3.78*	**13.2**	*3.78*	*16*
PFDoA	Not observed	*0.468*	Not observed	*0.468*	**1.45**	*0.468*	*30*
PFHpA	**176**	*0.877*	**44.3**	*0.877*	**39.7**	*0.877*	*18*
PFHxA	**379**	*20.0*	**258**	*20.0*	**145**	*20.0*	*11*

Table 8. OBSERVED PFAS CONCENTRATIONS, R.L. AND EMU VALUES FOR SAMPLES VP1364-07, VP1364-08, AND VP1364-10

Analyte	*Analyte Concentration (ng/L)*						EMU (±%)
	VP1364-07		VP1364-08		VP1364-10		
	Sample	*R.L.*	Sample	*R.L.*	Sample	*R.L.*	
PFNA	**1,210**	*20.0*	**59.1**	*2.20*	**13.1**	*2.20*	*21*
PFOA	**907**	*20.0*	**84.5**	*20.0*	**136**	*20.0*	*11*
PFPeA	**5,850**	*20.0*	**1,510**	*20.0*	**434**	*20.0*	*8.9*
PFUnA	**< R.L.**[1]	*5.20*	Not observed	*5.20*	Not observed	*5.20*	*30*
GenX	**190**	*20.0*	**2,030**	*20.0*	**97.6**	*20.0*	*19*
PFECA-A	**108**	*20.0*	**792**	*20.0*	**73.7**	*20.0*	*16*
PFECA-B	**0.756**	*0.676*	**2.96**	*0.676*	**< R.L.**[1]	*0.676*	*29*
PFECA-G	**4.04**	*0.974*	**9.71**	*0.974*	**2.84**	*0.974*	*46*
FOSA	**22.6**	*2.93*	**24.0**	*2.93*	**33.6**	*2.93*	*26*
N-EtFOSAA	**11.1**	*0.208*	**79.7**	*0.208*	**163**	*0.208*	*46*
N-EtFOSE	**1.16**	*0.208*	**1.94**	*0.208*	**3.23**	*0.208*	*37*
N-MeFOSE	**< R.L.**[1]	*2.64*	Not observed	*2.64*	Not observed	*2.64*	*20*
PFBS	**1,530**	*886*	**1,270**	*886*	**400**	*17.7*	*14*
PFDS	Not observed	*0.482*	**3.30**	*0.482*	**8.75**	*0.482*	*69*
PFHpS	**201**	*0.419*	**14.3**	*0.419*	**77.5**	*0.419*	*77*
$PFHxS_{Total}$	**1,610**	*32.0*	**138**	*32.0*	**224**	*32.0*	*26*
PFNS	**13.2**	*0.192*	Not observed	*0.192*	**9.68**	*0.192*	*57*
$PFOS_{Total}$	**24,400**	*926*	**4,830**	*926*	**5,120**	*926*	*45*
PFPeS	**37.7**	*18.8*	**18.5**	*0.590*	**26.3**	*18.8*	*17*
4:2 FTS	**< R.L.**[1]	*0.339*	**< R.L.**[1]	*0.339*	**< R.L.**[1]	*0.339*	*13*
6:2 FTS	**1.70**	*1.09*	**5.01**	*1.09*	**1.60**	*1.09*	*30*
8:2 FTS	**0.506**	*0.292*	Not observed	*0.292*	Not observed	*0.292*	*25*
10:2FTS	Not observed	*0.455*	**< R.L.**[1]	*0.455*	**< R.L.**[1]	*0.455*	*65*

Boldfaced underlined analytes are listed in the facility's NPDES permit for outfall 001
ng/ L= nanograms per liter
EMU = estimated measurement uncertainty
[1] < R.L.= analyte was observed at a concentration level less than its reporting limit

Table 9. OBSERVED PFAS CONCENTRATIONS, R.L. AND EMU VALUES FOR SAMPLES VP1364-12, VP1364-13, AND VP1364-14

Analyte	*Analyte Concentration (ng/L)*						EMU (±%)
	VP1364-12		VP1364-13		VP1364-14		
	Sample	*R.L.*	Sample	*R.L.*	Sample	*R.L.*	
PFBA	**27,400**	*1,000*	**22,600**	*1,000*	**30,800**	*1,000*	15
PFDA	**151**	*3.78*	**155**	*3.78*	**150**	*3.78*	*16*
PFDoA	Not observed	*0.468*	Not observed	*0.468*	Not observed	*0.468*	*30*
PFHpA	**132**	*0.877*	**125**	*0.877*	**135**	*0.877*	*18*
PFHxA	**315**	*20.0*	**312**	*20.0*	**359**	*20.0*	*11*
PFNA	**684**	*20.0*	**684**	*20.0*	**675**	*20.0*	*21*
PFOA	**544**	*20.0*	**599**	*20.0*	**605**	*20.0*	*11*

Table 9. OBSERVED PFAS CONCENTRATIONS, R.L. AND EMU VALUES FOR SAMPLES VP1364-12, VP1364-13, AND VP1364-14

Analyte	*Analyte Concentration (ng/L)*						EMU (±%)
	VP1364-12		VP1364-13		VP1364-14		
	Sample	*R.L.*	Sample	*R.L.*	Sample	*R.L.*	
PFPeA	**3,690**	*20.0*	**3,720**	*20.0*	**3,900**	*20.0*	*8.9*
PFUnA	Not observed	*5.20*	**< R.L.**[1]	*5.20*	Not observed	*5.20*	*30*
GenX	**818**	*20.0*	**761**	*20.0*	**1,010**	*20.0*	*19*
PFECA-A	**355**	*20.0*	**303**	*20.0*	**417**	*20.0*	*16*
PFECA-B	**1.67**	*0.676*	**1.11**	*0.676*	**2.02**	*0.676*	*29*
PFECA-G	**5.36**	*0.974*	**4.25**	*0.974*	**6.10**	*0.974*	*46*
FOSA	**14.1**	*2.93*	**17.5**	*2.93*	**15.2**	*2.93*	*26*
N-EtFOSAA	**7.82**	*0.208*	**8.82**	*0.208*	**6.12**	*0.208*	*46*
N-EtFOSE	Not observed	*0.208*	Not observed	*0.208*	Not observed	*0.208*	*37*
N-MeFOSE	Not observed	*2.64*	Not observed	*2.64*	Not observed	*2.64*	*20*
PFBS	**3,930**	*886*	**3,440**	*886*	**4,350**	*886*	*14*
PFDS	Not observed	*0.482*	Not observed	*0.482*	Not observed	*0.482*	*69*
PFHpS	**119**	*0.419*	**123**	*0.419*	**134**	*0.419*	*77*
$PFHxS_{Total}$	**772**	*32.0*	**1,070**	*32.0*	**895**	*32.0*	*26*
PFNS	Not observed	*0.192*	**13.2**	*0.192*	Not observed	*0.192*	*57*
$PFOS_{Total}$	**12,600**	*926*	**15,200**	*926*	**19,600**	*926*	*45*
PFPeS	**158**	*0.590*	**41.8**	*18.8*	**59.1**	*18.8*	*17*
4:2 FTS	Not observed	*0.339*	**< R.L.**[1]	*0.339*	**< R.L.**[1]	*0.339*	*13*
6:2 FTS	**15.8**	*1.09*	**13.2**	*1.09*	**18.5**	*1.09*	*30*
8:2 FTS	Not observed	*0.292*	Not observed	*0.292*	**0.604**	*0.292*	*25*
10:2FTS	Not observed	*0.455*	**< R.L.**[1]	*0.455*	Not observed	*0.455*	*65*

Boldfaced underlined analytes are listed in the facility's NPDES permit for outfall 001
ng/L= nano grams per liter
EMU= estimated measurement uncertainty
[1] < R.L.= Analyte was observed at a concentration level less than its reporting limit

Table 10. OBSERVED PFAS CONCENTRATIONS, R.L. AND EMU VALUES FOR SAMPLES VP1364-15, VP1364-16, VP1364-17, AND VP1364-18

Analyte	*Observed Analyte Concentration (ng/L)*								EMU (±%)
	VP1364-15		VP1364-16		VP1364-17		VP1364-18		
	Sample	*R.L.*	Sample	*R.L.*	Sample	*R.L.*	Sample	*R.L.*	
PFBA	**233,000**	*1,000*	**351,000**	*1,000*	**1,090,000**	*1,000*	**41,400**	*1,000*	15
PFDA	**125**	*3.78*	**151**	*3.78*	**152**	*3.78*	**175**	*3.78*	*16*
PFDoA	**10.6**	*0.468*	Not observed	*0.468*	Not observed	*0.468*	Not observed	*0.468*	*30*
PFHpA	**407**	*0.877*	**330**	*0.877*	**445**	*0.877*	**242**	*0.877*	*18*
PFHxA	**1,650**	*20.0*	**934**	*20.0*	**1,280**	*20.0*	**964**	*20.0*	*11*
PFNA	**599**	*20.0*	**864**	*20.0*	**577**	*20.0*	**> 515**[1]	*2.20*	*21*
PFOA	**778**	*20.0*	**718**	*20.0*	**587**	*20.0*	**829**	*20.0*	*11*
PFPeA	**9,390**	*20.0*	**8,460**	*20.0*	**10,500**	*1,000*	**7,220**	*20.0*	*8.9*
PFUnA	Not observed	*5.20*	Not observed	*5.20*	**< R.L.**[2]	*5.20*	Not observed	*5.20*	*30*

Table 10. OBSERVED PFAS CONCENTRATIONS, R.L. AND EMU VALUES FOR SAMPLES VP1364-15, VP1364-16, VP1364-17, AND VP1364-18

Analyte	*Observed Analyte Concentration (ng/L)*								EMU (±%)
	VP1364-15		VP1364-16		VP1364-17		VP1364-18		
	Sample	*R.L.*	Sample	*R.L.*	Sample	*R.L.*	Sample	*R.L.*	
GenX	**7,110**	*20.0*	**3,040**	*20.0*	**4,660**	*20.0*	**777**	*20.0*	*19*
PFECA-A	**2,460**	*20.0*	**2,220**	*20.0*	**9,791**	*20.0*	**219**	*20.0*	*16*
PFECA-B	Not observed	*0.676*	**9.12**	*0.676*	**36.0**	*0.676*	**1.31**	*0.676*	*29*
PFECA-G	Not observed	*0.974*	**21.4**	*0.974*	**69.2**	*0.974*	**5.75**	*0.974*	*46*
FOSA	**11.8**	*2.93*	**13.7**	*2.93*	**5.48**	*2.93*	**13.0**	*2.93*	*26*
N-EtFOSAA	Not observed	*0.208*	Not observed	*0.208*	Not observed	*0.208*	Not observed	*0.208*	*46*
N-EtFOSE	Not observed	*0.208*	Not observed	*0.208*	Not observed	*0.208*	Not observed	*0.208*	*37*
N-MeFOSE	Not observed	*2.64*	Not observed	*2.64*	Not observed	*2.64*	**40.8**	*2.64*	*20*
PFBS	**38,000**	*886*	**109,000**	*886*	**22,600**	*886*	**55,500**	*886*	*14*
PFDS	Not observed	*0.482*	Not observed	*0.482*	Not observed	*0.482*	Not observed	*0.482*	*69*
PFHpS	**316**	*0.419*	**101**	*0.419*	**356**	*0.419*	**87.8**	*0.419*	*77*
$PFHxS_{Total}$	**2,140**	*32.0*	**1,380**	*32.0*	**1,210**	*32.0*	**2,260**	*32.0*	*26*
PFNS	**19.5**	*0.192*	Not observed	*0.192*	Not observed	*0.192*	Not observed	*0.192*	*57*
$PFOS_{Total}$	**11,600**	*926*	**> 4,630**[2]	*18.5*	**9,470**	*926*	**> 417**[2]	*3.30*	*45*
PFPeS	**346**	*18.8*	**355**	*18.8*	**563**	*18.8*	**75.3**	*18.8*	*17*
4:2 FTS	Not observed	*0.339*	Not observed	*0.339*	Not observed	*0.339*	Not observed	*0.339*	*13*
6:2 FTS	**155**	*1.09*	**1.71**	*1.09*	Not observed	*1.09*	**1.98**	*1.09*	*30*
8:2 FTS	Not observed	*0.292*	Not observed	*0.292*	**2.61**	*0.292*	Not observed	*0.292*	*25*
10:2FTS	**4.02**	*0.455*	Not observed	*0.455*	**0.816**	*0.455*	Not observed	*0.455*	*65*

Boldfaced underlined analytes are listed in the facility's NPDES permit for outfall 001
ng/L= nano grams per liter
EMU= estimated measurement uncertainty
[1] <R.L.= analyte was observed at a concentration level less than its reporting limit
[2] ">" value indicated the associated response of the reported value was greater than the highest calibration standard solution

LC/MS QTOF ANALYSIS

In addition to the LC/MS QQQ quantification of PFAS, the 18 water samples were analyzed for presence of 34 PFAS listed in **Table 11**. The internal standard, mass-labelled GenX, was analyzed with the samples to show system repeatability and optimization. NEIC purchased reference materials for seven of the compounds listed in **Table 11**, and received reference materials for 22 of the compounds from 3M on February 13, 2020. No reference material was available for two of the compounds. **Table 12** shows the qualitative identifications of PFAS that were observed in the samples based on exact mass, retention time, adducts and dimers present, and fragmentation patterns. **Table 11** shows the target analytes, the abbreviations used throughout the analysis, and the formula for each analyte.

Table 11. TARGET ANALYTES, ABBREVIATIONS, AND FORMULAS		
Analyte Name	**Abbreviation**	**Formula**
4,4'-(Hexafluoroisopropylidene) diphenol[1]	4,4'-(Hexafluoroisopropylidene) diphenol	$C_{15}H_{10}F_6O_2$
4,8-dioxa-3H-perfluorononanoic acid[1, 2] and 4,8-dioxa-3H-perfluoro-3-(3-methoxy-propoxy)propanoic acid (sodium salt) [1]	ADONA NaDONA	$C_7H_2F_{12}O_4$ $C_7HF_{12}O_4Na$
Bis(perflourobutanesulfonyl)imide[1]	DBI-K salt	$C_8F_{18}NO_4S_2K$
Perfluorobutane sulfonamide (C4 amide) 1,1,2,2,3,3,4,4,4-Nonafluorobutane-1-sulfonamide[1, 2]	FBSA	$C_4H_2F_9NO_2S$
Perfluorobutyl sulfonamido acetic acid[1]	FBSAA (1)	$C_6H_3F_9NO_4SH$
[(Nonafluorobutane-1-sulfonyl)-carboxymethylamino] acetic acid[1]	FBSEE diacid	$C_8H_6F_9NO_6S$
Nonafluoro-N,N-bis(2-hydroxyethyl)butane-1- sulfonamide[1]	FBSEE diol	$C_8H_{10}F_9NO_4S$
Perfluoro-1-decanesulfonamide[2]	FDSA	$C_{10}H_2F_{21}NO_2S$
TRC19-0091 (2-Fluoromalonic Acid) [1]	Fluoropropanedioic acid	$C_3H_3FO_4$
Undecafluoro-2-methyl-3-oxahexanoic acid[1, 2]	GenX	$C_6HF_{11}O_3$
Nonafluoro-N-(2-hydroxyethyl)butane-1-sulfonamide[1]	HFBSE alcohol = FBSE	$C_6H_6F_9NO_3S$
Bis(trifluoromethylsulfonyl)amine[1]	HQ-115	$C_2F_6NO_4S_2H$
1,1,2,2,3,3,4,4,4-nonafluoro-N-methyl-1-Butanesulfonamide[1, 2]	MeFBSA	$C_5H_4F_9NO_2S$
Perfluorobutyl-methyl sulfonamido acetic acid[1]	MeFBSAA	$C_7H_6F_9NO_4S$
N-(2-Hydroxyethyl)perfluorobutanesulfonamide[1]	MeFBSE-OH	$C_7H_8F_9NO_3S$
1-Butanesulfonamide, N-(3-(dimethylamino)propyl)-1,1,2,2,3,3,4,4,4-nonafluoro-[1]	PBSA	$C_9H_{13}F_9N_2O_2S$
3-{[3-Dimethylamino)propyl](nonafluorobutane-1-sulfonyl)amino}propanoic acid[1]	PBSA-C1	$C_{12}H_{17}F_9O_4N_2S$
Perfluorobutanesulfinic Acid[1]	PFBSi	$C_4HF_9O_2S$
2,2,3,3-tetrafluoro-3-(trifluoromethoxy)-propanoic acid[2]	PFECA F or PMPA	$C_4HF_7O_3$
Perfluoro(2-ethoxyethane)sulfonate[2]	PFEESA	$C_4F_9O_4SH$
Perfluoroethanesulfonate[1]	PFES	$C_2HF_5O_3S$
2-[1-[difluoro(1,2,2,2-tetrafluoroethoxy)methyl]-1,2,2,2-tetrafluoroexthoxy]-1,1,2,2-tetrafluoro-ethanesulfonic acid[3]	PFESA (Nafion) Byproduct 2	$C_7H_2F_{14}O_5S$
Perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid[3]	PFESA Byproduct 1	$C_7HF_{13}O_5S$
2,2-difluoro-2-(trifluoromethoxy) acetic acid[3]	PFMOAA	$C_3HF_5O_3$
Perfluoro-3,5-dioxahexanoic acid[3]	PFO2HxA	$C_4HF_7O_4$
Perfluoro-3,5,7-trioxaoctanoic acid[3]	PFO3OA	$C_5HF_9O_5$
Perfluoro-3,5,7,9-tetraoxadecanoic acid[3]	PFO4OA	$C_6HF_{11}O_6$
Perfluoro-3,5,7,9, 11-pentadodecanoic acid[3]	PFO5OA or TAFN4	$C_7HF_{13}O_7$
Perfluoropropanesulfonate[3]	PFPS	$C_3F_7O_3SH$
Trifluoroacetic acid[1]	TFA	$C_2HF_3O_2$
Trifluoromethanesulfonate Lithium Triflate[1]	TFMS	CF_3O_3SH CF_3O_3SLi
Potassium 2,2,3,3-Tetrafluoropropionate[1] and 2,2,3,3-tetrafluoropropanoate	TFPA TFPAA	$C_3HF_4O_2K$ $C_3H_2F_4O_2$
[1]-Reference material received from 3M on February 13, 2020. [2]-Reference material currently available. [3]-Reference material currently unavailable.		

NEIC also received reference materials for perfluoroisobutyl amide (PIBA), propenoic acid 2-[methyl[(nonafluorobutyl)sulfonyl]amino]ethyl ester (N-MeFBSEA), and PBSA-DC from 3M. Standard solutions containing these three analytes were injected into the QTOF system with the samples, but were not observed using the current instrument conditions.

All analyses were performed using negative ion mode electrospray. For several compounds, the negative ion could be associated with either the acid or salt forms of the compound. For example, the anion identifying ADONA is the same anion as the sodium salt, NaDONA, and these QTOF analyses would not differentiate between the two compounds.

Table 12 shows the qualitative identification of the PFAS that were observed in the samples based on accurate and exact mass comparisons, retention time, adducts and dimers present, and fragmentation patterns. Tentative identifications were made for PFESA Byproduct 1 and PFPS, as reference materials needed for comparison purposes were not available for these analytes. All reported results were based on the anion form of the analyte presented in **Table 11**. Other PFAS present in the samples were reported using the LC/MS QQQ system. The internal standard, mass-labelled GenX, was identified in each sample injection to demonstrate that the instrumentation was operating as expected and in control. None of the PFAS listed in Table 12 are listed in the facility's NPDES permit for outfall 001.

Table 12. TENTATIVE IDENTIFICATION OF TARGET ANALYTES

Compound Name/Acronym	Sample No.																	
	VP1364-01	VP1364-02	VP1364-03	VP1364-04	VP1364-05	VP1364-06	VP1364-07	VP1364-08	VP1364-09	VP1364-10	VP1364-11	VP1364-12	VP1364-13	VP1364-14	VP1364-15	VP1364-16	VP1364-17	VP1364-18
4,4'-(Hexafluoro-isopropylidene)diphenol	---	---	---	---	---	---	---	***X***	---	---	---	***X***	***X***	***X***	***X***	---	---	---
ADONA	---	---	---	---	---	---	---	***X***	---	---	---	---	---	---	---	---	---	---
FBSA	---	---	---	---	---	---	***X***	***X***	---	***X***	---	***X***	***X***	***X***	---	***X***	***X***	***X***
FBSAA (1)	---	---	---	---	---	---	***X***	---	---	***X***	---	---	---	---	***X***	---	---	---
FBSEE diol	---	---	---	---	---	---	---	***X***	---	---	---	---	---	---	---	---	---	---
Gen-X	---	---	---	***X***	***X***	***X***	***X***	***X***	---	***X***	---	***X***	***X***	***X***	***X***	***X***	***X***	***X***
HFBSE Alcohol = FBSE	---	---	---	---	---	---	---	---	---	***X***	---	---	---	---	---	---	---	---
HQ-115[a]	---	---	---	---	---	---	***X***	***X***	---	***X***	---	***X***	***X***	***X***	***X***	***X***	---	***X***
PBSA	---	---	---	---	---	---	---	***X***	---	---	---	***X***	***X***	***X***	***X***	***X***	---	---
PBSA-C1	---	---	---	---	---	---	---	---	---	---	---	---	---	---	***X***	---	---	---
PFBSi	---	---	---	---	---	---	---	***X***	---	***X***	---	***X***	***X***	***X***	***X***	***X***	***X***	***X***
PFECA F or PMPA	---	---	---	---	---	---	***X***	---	---	---	---	---	---	---	---	---	---	**X**
PFEESA	---	---	---	---	---	---	***X***	***X***	---	***X***	---	***X***	***X***	***X***	***X***	***X***	***X***	***X***
PFES	---	---	---	---	---	---	***X***	***X***	---	***X***	---	***X***	***X***	***X***	---	***X***	***X***	***X***
PFESA Byproduct 1	---	---	---	√	---	---	---	√	---	---	---	√	√	√	√	√	---	√
PFPS	---	---	---	---	---	---	---	√	---	√	---	√	√	√	---	---	√	√
TFMS	---	---	---	---	---	---	---	---	---	---	---	***X***	***X***	***X***	---	---	---	---

X **–** Identified in the sample based on comparison to reference material.
√- Indicates a tentative identification of the analyte in which its mass spectra contained at least one ion whose observed accurate mass value was within 10 parts per million (ppm) of the exact mass value and had the appropriate fragments or adducts at the correct retention time. No response comparison to a reference solution was performed.
--- Indicates the analyte was not observed in the samples.
[a] DBI-K salt was observed in the blanks and will not be reported in the samples. HQ-115 was observed in the blanks but further investigation using MassHunter Quantitative software showed a background response in some blanks and substantially more in several samples. Those samples have been reported.

The criteria used for confirmation of the compounds reported in **Table 12** was that the compounds exhibited responses greater than the low standard solution (equal to or greater

than 10 ng/mL) for that respective compound. Taking into account the sample extraction, these analyte concentrations would be greater than 20 ng/L. For PFECA F, the low standard was 50 ng/mL, which would be greater than 100 ng/L in the sample. PFPS and PFESA Byproduct 1 are tentative with no related standard, so the relative concentration in the sample cannot be determined.

INVESTIGATION OBSERVATIONS

NEIC made the following observations during the multimedia compliance inspection. NEIC field team members discussed all observations with facility representatives during the closeout meeting unless otherwise noted in the observation description below.

These observations are not final compliance determinations. EPA OCE and Region 5 will make the final compliance determinations based on its review of this report and other technical, regulatory, and facility information.

Observation: CWA 1
Observation Summary: 3M Cordova was not documenting the time between sample collection and sample analysis for pH grab samples at outfalls A01 and 001; therefore, sample holding times could not be confirmed.
Citation: **NPDES Permit No. IL0003140, Effluent Limitations and Monitoring, Outfalls 001, A01** – *From the effective date of this permit until the expiration date, the effluent of the following discharges shall be monitored and limited at all times as follows…* *Outfall 001 – pH, daily grab samples* *Outfall A01 – pH, 4/week grab samples* **40 Code of Federal Regulations (CFR) § 136.3, Table II – Required Containers, Preservation Techniques, and Holding Times** – *Hydrogen ion (pH), maximum holding time – analyze within 15 minutes*
Evidence: **Appendix CWA A** – NPDES Permit No. IL0003140 **Appendix CWA E** – Waste Treatment Data Sheet
Description of Observation: 3M Cordova is required by its NPDES permit to monitor for pH at outfall 001 (daily grab sample) and outfall A01 (grab sample 4 times per week). According to 3M Cordova WWTP operators, the grab samples are collected at the respective outfall locations every 2 hours and returned to building 14 for pH analysis using a bench-top pH meter. Analytical results for pH samples are logged on a "Waste Treatment Data Sheet" (**Appendix CWA E**). The time between sample collection and sample analysis for the pH samples is not documented; therefore, the sample holding times (i.e., within 15 minutes) could not be confirmed.

Observation: CWA 2
Observation Summary: The sampling location used by 3M Cordova for monitoring at outfall A01 was not representative of the discharge. In addition, the sample tubing used as part of sample collection at outfall A01 was not being properly maintained.
Citation: **NPDES Permit No. IL0003140, Effluent Limitations and Monitoring, Outfall A01** – *From the effective date of this permit until the expiration date, the effluent of the following discharges shall be monitored and limited at all times as follows…*

Observation: CWA 2

NPDES Permit No. IL0003140, Special Condition 5 – *Samples taken in compliance with the internal monitoring requirements for Internal Outfall A01 shall be taken at a point representative of the discharge but prior to mixing with the discharge of non-contact cooling water from Outfall 001.*

NPDES Permit No. IL0003140, Standard Conditions (5) Proper operation and maintenance. *The permittee shall at all times properly operate and maintain all facilities and systems of treatment and control (and related appurtenances) which are installed or used by the permittee to achieve compliance with the conditions of this permit. Proper operation and maintenance also includes adequate laboratory controls and appropriate quality assurance procedures. This provision requires the operation of back-up or auxiliary facilities or similar systems which are installed by a permittee only when the operation is necessary to achieve compliance with the conditions of the permit.*

Evidence:
Appendix CWA A – NPDES Permit No. IL0003140
Appendix CWA B – NEIC CWA Inspection Photographs

Description of Observation:

3M Cordova is required by its NPDES permit to monitor for several parameters at outfall A01 by grab or composite sample collection. Outfall A01 is the discharge of treated wastewater from the 3M Cordova WWTP before it is mixed with noncontact cooling water from the facility. The last unit in the WWTP is pond #3. Wastewater from pond #3 flows by gravity through an outlet structure located in the northeast corner of the pond and over a V-notch weir. Wastewater discharged over the weir enters a channel and flows approximately 30 feet before flowing into the main facility discharge channel. The wastewater discharge from outfall A01 combines with facility NCCW in the main facility discharge channel, and the combined wastewater and NCCW is then ultimately discharged to the Mississippi River via outfall 001.

3M Cordova collects samples at outfall A01 for NPDES permit compliance by using a submersible pump located in the outlet structure of the pond, upstream of the discharge weir, to pump wastewater through tubing into a building called the "Discharge to River" (DTR) shack (**Appendix CWA B**, photographs RIMG0037, RIMG0038, and RIMG0039). The sample tubing from the pump is connected to piping that eventually empties into a sink in the DTR shack. 3M Cordova collects grab samples from the wastewater flowing into the sink, and collects composite samples manually, by collecting grab samples from the wastewater flowing into the sink every 2 hours over a 24-hour period and then combining the grab sample aliquots.

Because samples for outfall A01 are collected using a submersible pump located in the outlet structure of the pond upstream of the discharge weir, and the wastewater is collected from the bottom depth of the pond, the samples collected at outfall A01 are not representative of the discharge.

At the time of the inspection, the sample tubing from the submersible pump used for sample collection at outfall A01 had visible solids deposition, which may cause sample result bias (**Appendix CWA B**, photograph RIMG0038).

Observation: CWA 3

Observation Summary: 3M Cordova's procedure for sampling at stormwater outfalls 003 and 004 may result in the reporting of data on discharge monitoring reports (DMRs) that are not representative of discharge conditions.

Citation:

NPDES Permit No. IL0003140, Effluent Limitations and Monitoring, Outfalls 003, 004 – *From the effective date of this permit until the expiration date, the effluent of the following discharges shall be monitored and limited at all times as follows…*

*Flow, 1/Quarter**, Measure*
126 Priority Pollutants, 1/Year, Composite
*Iron (Total), 1/Quarter**, Composite*

***Monitor during the months of March, June, September, and December*

NPDES Permit No. IL0003140, Special Condition 6 – *Samples taken in compliance with the effluent monitoring requirements for Outfalls 001, 002, 003, and 004 shall be taken at a point representative of the discharges, but prior to entry into the receiving stream.*

Evidence:

Appendix CWA A – NPDES Permit No. IL0003140
Appendix CWA B – NEIC CWA Inspection Photographs

Description of Observation:

3M Cordova manages stormwater runoff collected for discharge through outfalls 003 and 004 by storing it behind a normally closed sluice gate prior to its discharge (**Appendix CWA B**, photographs RIMG0041 and RIMG0043). According to 3M Cordova representatives, not all storm events require the sluice gates to be opened and discharge stormwater through outfalls 003 and 004. 3M Cordova's procedure for NPDES permit compliance sampling at outfalls 003 and 004 is to collect samples from the stormwater collected behind the closed sluice gate (i.e., during conditions of no discharge). Those samples are analyzed, and the sample results are reported for outfalls 003 and 004 on DMRs, even in instances when the sluice gate remains closed and no discharge occurs. In some instances, 3M Cordova noted on the DMR that sample results are reported but there was no discharge (e.g., first quarter 2019 DMR for outfall 004).

Observation: CWA 4

Observation Summary: NEIC identified issues related to composite sample monitoring requirements in 3M Cordova's NPDES permit during the inspection.

Citation:

NPDES Permit No. IL0003140, Effluent Limitations and Monitoring, Outfalls 001, A01, 002, 003, 004 – *From the effective date of this permit until the expiration date, the effluent of the following discharges shall be monitored and limited at all times as follows…*

NPDES Permit No. IL0003140, Standard Conditions, Definitions.

> ***24-Hour Composite Sample*** *means a combination of at least 8 sample aliquots of at least 100 milliliters, collected at periodic intervals during the operating hours of a facility over a 24-hour period.*

Observation: CWA 4

8-Hour Composite Sample *means a combination of at least 3 sample aliquots of at least 100 milliliters, collected at periodic intervals during the operating hours of a facility over an 8-hour period.*

Flow Proportional Composite Sample *means a combination of sample aliquots of at least 100 milliliters collected at periodic intervals such that either the time interval between each aliquot or the volume of each aliquot is proportional to either the stream flow at the time of sampling or the total stream flow since the collection of the previous aliquot.*

Evidence:

Appendix CWA A – NPDES Permit No. IL0003140

Description of Observation:

The monitoring requirements in 3M Cordova's NPDES permit only specify the collection of a "composite" sample for several parameters at outfalls 001, A01, 002, 003, and 004. The standard conditions of the permit include definitions and conditions for three types of composite samples: 24-hour composite sample, 8-hour composite sample, and flow proportional composite sample. However, the permit does not specify the type of composite sample to be collected. 3M Cordova representatives stated they are collecting composite samples at the outfalls by collecting sample aliquots (approximately 250 milliliters or 475 milliliters depending on sample location) every 2 hours over a 24-hour period and manually compositing the sample aliquots. The composite samples are not flow-proportioned.

3M Cordova's NPDES permit requires cyanide to be monitored at outfalls 001 and A01 by collection of a composite sample. Because cyanide is very reactive and unstable and requires special preservation techniques for sample collection, grab samples, rather than composite samples, are typically required to be collected.

For example, the NPDES permit application requirements under 40 CFR § 122.21 (g)(7) (Application requirements for existing manufacturing, commercial, mining, and silvicultural dischargers) contain the following language for samples required to be collected for application purposes:

…When paragraph (g)(7) of this section requires analysis of pH, temperature, cyanide, total phenols, residual chlorine, oil and grease, fecal coliform (including E. coli), and Enterococci (previously known as fecal streptococcus at §122.26 (d)(2)(iii)(A)(3)), or volatile organics, grab samples must be collected for those pollutants….

Observation: CWA 5

Observation Summary: NEIC inspectors observed slack in the sample collection tubing on automatic composite samplers 3M Cordova used at outfalls 001 and A01 for PFAS characterization, potentially resulting in incomplete evacuation of sample rinses between sample aliquots. In addition, not enough information was available to determine all the types of materials 3M Cordova was using in the sampling of outfalls A01 and 001 for PFAS, whether for NPDES permit compliance sampling or for PFAS characterization sampling.

Citation:

None

Observation: CWA 5

Evidence:
Appendix CWA B – NEIC CWA Inspection Photographs
Inspection observations

Description of Observation:

During the NEIC inspection, 3M Cordova staff stated that automatic composite wastewater samplers had been installed a week or two prior to the inspection to begin PFAS characterization sampling. The automatic composite samplers were installed at the production wellfield (building 10), the nickel/fluoride (Ni/F) clarifier, outfall A01, and outfall 001.

The automatic composite samplers at outfalls A01 and 001 used for characterization sampling were not being used for NPDES permit compliance sampling, according to 3M Cordova representatives; however, the automatic samplers were drawing samples from the same location 3M Cordova uses for NPDES permit compliance sampling. As described above, 3M Cordova collects samples at outfall A01 for NPDES permit compliance by using a submersible pump located in the outlet structure of the pond to pump wastewater through tubing into the DTR shack. The sample tubing from the pump is connected to piping that eventually empties into a sink in the DTR shack. In similar fashion, the discharge from outfall 001 is pumped through piping into the DTR shack and into an adjacent sink (**Appendix CWA B**, photographs RIMG0037, RIMG0038, and RIMG0039).

For the PFAS characterization sampling, automatic composite samplers were placed on the floor in the DTR shack (one each for outfalls A01 and 001). The sample collection tubing for each sampler ran up and over from the samplers into the respective sinks for each outfall and was taped into a cup in the bottom of each sink. The water that collected in each cup was pumped/suctioned through the tubing back into the composite samplers (**Appendix CWA B**, photographs RIMG0039 and RIMG0040). NEIC inspectors observed slack in the composite sampler tubing and standing water in the tubing. Slack in the tubing can cause incomplete evacuation of sample rinses between sample aliquots (as evidenced by the standing water in the tubing). Incomplete evacuation of sample rinses between sample aliquots results in sample aliquots, and subsequently composite samples, that are not representative of the discharge being sampled over time.

While no Clean Water Act method currently exists for sampling and analyzing for PFAS in wastewater, EPA guidance includes information on precautions to take during PFAS sampling to prevent sample contamination or loss of the analyte, including prohibited materials and equipment to use during sampling, as well as recommended materials and equipment to use (*EPA PFAS Methods and Guidance for Sampling and Analyzing Water and Other Environmental Media, EPA/600/F-17/022f, updated June 2019*). For example, polytetrafluoroethylene materials and equipment (tubing, sample containers, tape, pumps, valves) contain PFAS and should not be used in sampling for PFAS. At the time of the NEIC inspection, not enough information was available to determine all the types of materials 3M Cordova was using in the sampling of outfalls A01 and 001 for PFAS, whether for NPDES permit compliance sampling or for PFAS characterization sampling.

Observation: CWA 6

Observation Summary: The stormwater construction permit lists best management practices (BMPs) from the state of California, not Illinois.

Citation:

NPDES Permit No. ILR10 Part IV D.2.Controls. *Each plan shall include a description of appropriate controls that will be implemented at the construction site and any off-site stockpile or storage area unless already authorized by a separate NPDES permit. The plan shall include details or drawings that show proper installation of controls and BMPs. The Illinois Urban Manual or other similar documents shall be used for developing the appropriate management practices, controls or revisions of the plan.*

Evidence:

Appendix CWA F – General NPDES Permit Storm Water Construction ILR10
Appendix CWA G – Storm Water Pollution Prevention Plan

Description of Observation:

Construction runoff from 3M Cordova is regulated under NPDES permit No. ILR10 (3M Cordova was assigned permit No. ILR10AJ08). The permit requires implementation of BMPs to control polluted runoff from the construction area. 3M Cordova has a storm water pollution prevention plan that addresses requirements for controlling runoff from construction activities on-site. The permit issued by the state of Illinois requires BMPs listed in the Illinois *Storm Water Management Manual* to be employed. The SWPPP for 3M Cordova lists BMPs from the state of California, not Illinois.

Observation: CWA 7

Observation Summary: Two stormwater outfalls not listed in the 3M Cordova NPDES permit, or in any other facility NPDES permit, were identified during the inspection during discussions with 3M Cordova staff and review of facility records.

Citation: NPDES Permit No. IL0003140, Effluent Limitations and Monitoring, Outfalls 002, 003, 004 – Stormwater Runoff

From the effective date of this permit until the expiration date, the effluent of the following discharges shall be monitored and limited at all times as follows…

Evidence:

Appendix CWA A – NPDES Permit No. IL0003140
Appendix CWA G – Storm Water Pollution Prevention Plan
Appendix CWA C – 2007 NPDES Permit Renewal Application
Appendix CWA D – 2017 NPDES Permit Renewal Application
Appendix CWA B – NEIC CWA Inspection Photographs
Inspection observations and discussions

Description of Observation:

3M Cordova's NPDES permit includes three listed and authorized stormwater outfalls (002, 003, and 004). During review of maps and records, NEIC also discovered that 3M Cordova has an outfall B (or outlet B) discussed in the SWPPP and included on site drainage maps, and an outlet A-1 included on site drainage maps (not included in the SWPPP), both located on the west side of the facility along the Mississippi River. (NEIC does not have electronic copies of site drainage maps due to the size of the maps, but does have hard copies available.) Neither outfall B nor outlet A-1 is included in 3M Cordova's current NPDES permit.

Observation: CWA 7

3M Cordova's 2007 NPDES permit renewal application (application for the current effective permit) and 2017 NPDES permit renewal application (application for the permit renewal yet to occur) both include only outfalls 002 (A), 003 (C), and 004 (D) listed on EPA Form 2F (**Appendices CWA C** and **CWA D**, pages 51 and 45, respectively). However, both permit renewal applications include a narrative reference in Attachment 2F-IVC to outfall B (**Appendix CWA C and CWA D**, pages 66 and 58, respectively): "The structural controls found in the drainage area for Outfall B include indoor storage of hazardous waste and raw materials and a gated structure that is closed to contain spills or contaminated storm water to allow transfer to the wastewater treatment plant, if necessary." Both permit renewal applications also state, "The gate to Outfall B is never opened." During the inspection, Keith Schmuck, 3M Cordova quality manager, stated that the gate at outfall B stays closed and impounded water behind the gate either percolates or evaporates. EPA inspectors observed outfall/outlet B on several occasions during the inspection (**Appendix CWA B**, photographs RIMG0044 and RIMG0055).

Outlet A-1 is not listed or mentioned in 3M Cordova's SWPPP or in the permit renewal applications. Outlet A-1 is shown on site drainage maps in the extreme northwest area of the facility along the Mississippi River. During the inspection on December 5, 2019, NEIC and 3M Cordova representatives attempted to locate outlet A-1; no discrete outlet or conveyance was found or observed. The location of outlet A-1 on the site drainage maps is a wooded area, with no observed industrial activity at the time of the inspection.

Observation: CWA 8

Observation Summary: Analytical results for samples collected by NEIC during the inspection of production wellfield water, and analyzed by the NEIC laboratory, show elevated concentrations of several PFAS. Production wellfield water is used for process water purposes and as once-through NCCW; most wellfield water (approximately 7 million gallons per day [MGD]) is discharged as untreated NCCW to the Mississippi River via outfall 001.

Citation: None

Evidence:

Appendix RCRA A - Environmental Assessment for Perfluorochemicals Summary Report
Appendix CWA B – NEIC CWA Inspection Photographs
Inspection observations and discussions
NEIC analytical data

Description of Observation:

Process and cooling source water are obtained from a wellfield located east of the 3M Cordova facility. The wellfield consists of seven wells (ranging from 117 to 183 feet deep), and the daily flow from the wellfield ranges from 7.2 MGD to 12.2 MGD. In 2014, Illinois EPA designated the groundwater under the facility a groundwater management zone, including the process wellfield area. 3M Cordova groundwater elevation measurements confirm the flow of groundwater to the east, away from the Mississippi River. 3M Cordova assessments and modeling show that the production wells are capturing the groundwater. Sludge from the 3M Cordova WWTP was historically land-incorporated from 1975 to 1999 in the area adjacent to the wellfield (east of Highway 84; see figure 3 on page 39 of **Appendix RCRA A**).

Observation: CWA 8

The state of Illinois still classifies this water supply system as a "Non-Transient, Non-Community Public Water System" (PWS ID No. IL3049031), which serves a population of 300. 3M Cordova personnel reported that the wells have been determined to be "Ground Water Under the Direct Influence of Surface Water." At the time of the inspection, this water source was not being used as a drinking water supply at the facility; however, it has been used for that purpose in the past.

NEIC collected water and wastewater samples from several locations throughout the 3M Cordova facility on December 6, 2020, including one sample collected from the production wellfield water (collected from a sample tap in building 10 from one of the two 250,000-gallon storage tanks at the wellfield, prior to being pumped to the facility) (**Appendix CWA B**, photograph RIMG0047). The sample is identified as VP1364-07 and was analyzed for numerous PFAS.

The following are the quantitative analytical results for sample VP1364-07, reported in concentration levels of ng/L (equivalent to parts per trillion [ppt]).

For reference purposes, in 2016 EPA established a Lifetime Health Advisory level in drinking water for PFOA and PFOS at 70 ppt, separately or combined. Also, Illinois EPA has proposed the following groundwater standards for five PFAS: PFBS (140,000 ppt), PFHxS (140 ppt), PFNA (21 ppt), PFOA (21 ppt), and PFOS (14 ppt), and a combined PFOA and PFOS standard of 21 ppt.

Analyte	**Sample VP1364-07** Production Wellfield Water **Analyte Concentration (ng/L)**
PFBA	10,800
PFDA	193
PFHpA	176
PFHxA	379
PFNA	1,210
PFOA	907
PFPeA	5,850
GenX	190
PFECA-A	108
PFECA-B	0.756
PFECA-G	4.04
FOSA	22.6
N-EtFOSAA	11.1
N-EtFOSE	1.16
PFBS	1,530
PFHpS	201
$PFHxS_{Total}$	1,610
PFNS	13.2
$PFOS_{Total}$	24,400
PFPeS	37.7
6:2 FTS	1.70
8:2 FTS	0.506

Observation: CWA 8

In addition to the quantitative analysis, NEIC performed a qualitative analysis of the samples for the presence of 34 PFAS. The following PFAS were observed in sample VP1364-07 (production wellfield water).

- FBSA (Perfluorobutane sulfonamide)
- FBSAA(1) (Perfluorobutyl sulfonamido acetic acid)
- GenX (Undecafluoro-2-methyl-3-oxahexanoic acid)
- HQ-115 (Bis(trifluoromethylsulfonyl)amine)
- PFECA F or PMPA (2,2,3,3-tetrafluoro-3-(trifluoromethoxy)-propanoic acid)
- PFEESA (Perfluoro(2-ethoxyethane)sulfonate)
- PFES (Perfluoroethanesulfonate)

Observation: CWA 9

Observation Summary: Analytical results for samples collected by NEIC during the inspection of impounded wastewater/stormwater at outfall 004 and outlet/outfall B, and analyzed by the NEIC laboratory, show elevated concentrations of several PFAS. 3M Cordova is not required by its NPDES permit to monitor for PFAS at stormwater outfalls.

Citation: None

Evidence:
Appendix CWA B – NEIC CWA Inspection Photographs
Inspection observations
NEIC analytical data

Description of Observation:

3M Cordova manages stormwater runoff collected for discharge through outfall 004 by storing it behind a normally closed sluice gate prior to its discharge. 3M Cordova manages stormwater runoff collected in the drainage area that contributes to outlet/outfall B by storing the stormwater behind a closed sluice gate. According to Keith Schmuck, 3M Cordova quality manager, the gate at outlet/outfall B stays closed, and impounded water behind the gate either percolates or evaporates.

NEIC collected water and wastewater samples from several locations throughout the 3M Cordova facility on December 6, 2020, including one sample collected from impounded wastewater/stormwater upstream of the closed sluice gate at outfall 004 (sample VP1364-08), and one sample collected from impounded wastewater/stormwater upstream of the closed sluice gate at outlet B (sample VP1364-10) (**Appendix CWA B**, photographs RIMG0051 and RIMG0055, respectively). No discharges from the facility stormwater outfalls occurred during the inspection, to NEIC's knowledge. No sample was collected at outfall 002 as no discharge occurred, and no sample was collected at outfall 003 as no discharge occurred and there was no impounded wastewater/stormwater at the outfall structure.

The following are the quantitative analytical results for samples VP1364-08 and VP1364-10, reported in concentration levels of ng/L (equivalent to ppt). 3M Cordova is not required by its NPDES permit to monitor for PFAS at stormwater outfalls. While the following results are of impounded wastewater/stormwater and not of actual discharge events, the elevated concentrations of PFAS in the impounded wastewater/stormwater provide an indication of PFAS levels in stormwater runoff from two facility drainage areas, that could be released and discharged to the Mississippi River or percolate into the groundwater.

Observation: CWA 9

Analyte	**Sample VP1364-08** Stormwater outfall 004 (sample collected from impounded wastewater/ stormwater upstream of the closed sluice gate, no discharge occuring at time of sample collection) **Analyte Concentration (ng/L)**	**Sample VP1364-10** Stormwater outlet/outfall B (sample collected from impounded wastewater/ stormwater upstream of the closed sluice gate, no discharge occuring at time of sample collection) **Analyte Concentration (ng/L)**
PFBA	36,800	6,490
PFDA	17.6	13.2
PFDoA	Not observed	1.45
PFHpA	44.3	39.7
PFHxA	258	145
PFNA	59.1	13.1
PFOA	84.5	136
PFPeA	1,510	434
GenX	2,030	97.6
PFECA-A	792	73.7
PFECA-B	2.96	Less than reporting limit
PFECA-G	9.71	2.84
FOSA	24.0	33.6
N-EtFOSAA	79.7	163
N-EtFOSE	1.94	3.23
PFBS	1,270	400
PFDS	3.30	8.75
PFHpS	14.3	77.5
$PFHxS_{Total}$	138	224
PFNS	Not observed	9.68
$PFOS_{Total}$	4,830	5,120
PFPeS	18.5	26.3
4:2 FTS	< R.L.	< R.L.
6:2 FTS	5.01	1.60

In addition to the quantitative analysis, NEIC performed a qualitative analysis of the samples for the presence of 34 PFAS. The following PFAS were observed in samples VP1364-08 and VP1364-10.

PFAS observed in sample VP1364-08

- 4,4'-(Hexafluoro-isopropylidene)diphenol
- ADONA (4,8-Dioxa-3H-perfluorononanoic acid)
- FBSA (Perfluorobutane sulfonamide (C4 amide) 1,1,2,2,3,3,4,4,4-Nonafluorobutane-1-sulfonamide)
- FBSEE diol (Nonafluoro-N,N-bis(2-hydroxyethyl)butane-1- sulfonamide)
- Gen-X (Undecafluoro-2-methyl-3-oxahexanoic acid)
- HQ-115 (Bis(trifluoromethylsulfonyl)amine)
- PBSA (1-Butanesulfonamide, N-(3-(dimethylamino)propyl)-1,1,2,2,3,3,4,4,4-nonafluoro-)
- PFBSi (Perfluorobutanesulfinic Acid)
- PFEESA (Perfluoro(2-ethoxyethane)sulfonate)
- PFES (Perfluoroethanesulfonate)
- PFESA Byproduct 1 (Perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid)

Observation: CWA 9

- PFPS ((Perfluoropropanesulfonate)

PFAS observed in sample VP1364-10

- FBSA (Perfluorobutane sulfonamide (C4 amide) 1,1,2,2,3,3,4,4,4-Nonafluorobutane-1-sulfonamide)
- FBSAA (1) (Perfluorobutyl sulfonamido acetic acid)
- Gen-X (Undecafluoro-2-methyl-3-oxahexanoic acid)
- HFBSE Alcohol = FBSE (Nonafluoro-N-(2-hydroxyethyl)butane-1-sulfonamide)
- HQ-115 (Bis(trifluoromethylsulfonyl)amine)
- PFBSi (Perfluorobutanesulfinic Acid)
- PFEESA (Perfluoro(2-ethoxyethane)sulfonate)
- PFES (Perfluoroethanesulfonate)
- PFPS ((Perfluoropropanesulfonate)

Observation: CWA 10

Observation Summary: Analytical results for wastewater samples collected by NEIC during the inspection at outfall 001, and analyzed by the NEIC laboratory, show the discharge of elevated concentrations of several PFAS to the Mississippi River and the discharge of several PFAS that are not listed in 3M Cordova's NPDES permit and, therefore, not monitored for in the discharge.

Citation:

40 CFR § 122.1(b) – *Scope of the NPDES permit requirement. (1) The NPDES program requires permits for the discharge of "pollutants" from any "point source" into "waters of the United States." The terms "pollutant", "point source" and "waters of the United States" are defined at §122.2.*

NPDES Permit No. IL0003140 – *In compliance with the provisions of the Illinois Environmental Protection Act, Title 35 of Ill. Adm. Code, Subtitle C and/or Subtitle D, Chapter 1, and the Clean Water Act (CWA), the above-named permittee is hereby authorized to discharge at the above location to the above-named receiving stream in accordance wth the standard conditions and attachments herein.*

NPDES Permit No. IL0003140, Effluent Limitations and Monitoring, Outfall 001 – *From the effective date of this permit until the expiration date, the effluent of the following discharges shall be monitored and limited at all times as follows…*

Parameter[*]	*Concentration Limits*	*Sample Frequency*	*Sample Type*
Perfluorobutanoic Acid (PFBA)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluoropentanoic Acid (PFPeA)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluorohexanoic Acid (PFHxA)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluoroheptanoic Acid (PFHpA)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluorooctanoic Acid (PFOA)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluorononanoic Acid (PFNA)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluorodecanoic Acid (PFDA)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluoroundecanoic Acid (PFUnA)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluorododecanoic Acid (PFDoA)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluorotridecanoic Acid (PFTrA)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluorobutanesulfanate (PFBS)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluoroheaxanesulfonate (PFHxS)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluorooctanesulfonate (PFOS)	*Monitor Only*	*1/Quarter*	*Grab*[**]
Perfluorooctanesulfonamide (FOSA)	*Monitor Only*	*1/Quarter*	*Grab*[**]

Observation: CWA 10

* PFAS acronyms are not listed in the 3M Cordova NPDES permit. Acronyms are listed here by NEIC for comparative purposes.
**Monitor during the months of March, June, September, and December

Evidence:
Appendix CWA A – NPDES Permit No. IL000314
Appendix CWA B – NEIC CWA Inspection Photographs
NEIC analytical data

Description of Observation:

3M Cordova is authorized to discharge through outfall 001 to the Mississippi River, with the discharge consisting of noncontact cooling water, treated wastewater (via internal outfall A01), and stormwater runoff. 3M Cordova is required in its NPDES permit to monitor quarterly for 14 PFAS (listed above) at outfall 001 by grab sample, with no effluent limitations (monitor only) specified in the permit for those parameters.

NEIC collected water and wastewater samples from several locations throughout the 3M Cordova facility on December 6, 2020, including three samples collected from the discharge at outfall 001, also known as the "Discharge to River" (DTR). The samples collected by NEIC at outfall 001 (DTR) are identified as VP1364-12, VP1364-13, and VP1364-14 (**Appendix CWA B**, photograph RIMG0059). The samples were analyzed by the NEIC laboratory for numerous PFAS.

The following are the quantitative analytical results for samples VP1364-12, VP1364-13, and VP1364-14, reported in concentration levels of ng/L (equivalent to ppt). Included are results for both those PFAS listed in the NPDES permit, and monitored for by 3M Cordova in the discharge, as well as those not listed in the NPDES permit and, therefore, not monitored for in the discharge (and are noted as such).

Analyte	Analyte Concentration (ng/L)		
	Sample VP1364-12 Outfall 001 (DTR)	**Sample VP1327-13** Outfall 001 (DTR)	**Sample VP1364-14** Outfall 001 (DTR)
PFBA[1]	27,400	22,600	30,800
PFDA[1]	151	155	150
PFHpA[1]	132	125	135
PFHxA[1]	315	312	359
PFNA[1]	684	684	675
PFOA[1]	544	599	605
PFPeA[1]	3,690	3,720	3,900
GenX[2]	818	761	1,010
PFECA-A[2]	355	303	417
PFECA-B[2]	1.67	1.11	2.02
PFECA-G[2]	5.36	4.25	6.10
FOSA[1]	14.1	17.5	15.2
N-EtFOSAA[2]	7.82	8.82	6.12
PFBS[1]	3,930	3,440	4,350
PFHpS[2]	119	123	134

Observation: CWA 10

$PFHxS_{Total}$[1]	772	1,070	895
PFNS[2]	Not observed	13.2	Not observed
$PFOS_{Total}$[1]	12,600	15,200	19,600
PFPeS[2]	158	41.8	59.1
6:2 FTS[2]	15.8	13.2	18.5

[1] PFAS included in the 3M Cordova NPDES permit at outfall 001. PFTrA was not detected in any sample.
[2] PFAS not included in the 3M Cordova NPDES permit.

In addition to the quantitative analysis, NEIC performed a qualitative analysis of the samples for the presence of 34 PFAS. The following PFAS were observed in samples VP1364-12, VP1364-13, and VP1364-14, none of which are listed in the NPDES permit or authorized for discharge.

- 4,4'-(Hexafluoroisopropylidene) diphenol
- FBSA (Perfluorobutane sulfonamide)
- GenX (Undecafluoro-2-methyl-3-oxahexanoic acid)
- HQ-115 (Bis(trifluoromethylsulfonyl)amine)
- PBSA (1-Butanesulfonamide, N-(3-(dimethylamino)propyl)-1,1,2,2,3,3,4,4,4-nonafluoro)
- PFBSi (Perfluorobutanesulfinic acid)
- PFEESA (Perfluoro(2-ethoxyethane)sulfonate)
- PFES (Perfluoroethanesulfonate)
- PFESA Byproduct 1 (Perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid)
- PFPS ((Perfluoropropanesulfonate)
- TFMS (Trifluoromethanesulfonate ion)

Observation: CWA 11

Observation Summary: Analytical results for samples collected by NEIC during the inspection of treated process wastewater at outfall A01 (pond #3), and analyzed by the NEIC laboratory, show elevated concentrations of several PFAS. Following the discharge of treated wastewater from outfall A01, no further treatment of the wastewater occurs, only the mixing of wastewater with facility NCCW and the subsequent discharge to the Mississippi River.

Citation: None

Evidence:
Inspection observations
NEIC analytical data

Description of Observation:

Pond #3 is the last treatment unit in 3M Cordova's on-site WWTP for the treatment of facility process wastewater, including the treatment of wastewater from the electronic materials factory (i.e., fluorochemical manufacturing) and facility thermal oxidizer. The discharge from pond #3 discharges over a V-notch weir through internal outfall A01. Wastewater discharged over the weir enters a channel and flows approximately 30 feet before flowing into the main facility discharge channel. The wastewater discharge from outfall A01 combines with facility NCCW in the main facility discharge channel, and the combined wastewater and NCCW is then ultimately discharged to the Mississippi River via outfall 001.

NEIC collected water and wastewater samples from several locations throughout the 3M Cordova facility on December 6, 2020, including one sample collected at outfall A01 from the

Observation: CWA 11

discharge stream directly downstream of the V-notch weir (sample VP1364-15). NEIC also collected untreated process wastewater samples from WWTP lift station B63 (influent to the nickel/fluoride treatment system at the on-site WWTP) (sample VP1364-16), building 30 and the thermal oxidizer discharge (sample VP1364-17), and building 20 discharge (sample VP1364-18) (**Appendix CWA B**, photographs RIMG0061, RIMG0064, RIMG0066 and RIMG0069, respectively).

The following are the quantitative analytical results for samples VP1364-15, VP1364-16, VP1364-17, and VP1364-18, reported in concentration levels of ng/L (equivalent to ppt). Samples VP1364-16, VP1364-17, and VP1364-18 provide a relative indication of PFAS concentrations of untreated process wastewater at various locations, while sample VP1364-15 shows PFAS concentrations following process wastewater treatment, but treatment not specifically designed for removing PFAS. Following the discharge of treated wastewater from outfall A01, no further treatment of the wastewater occurs, only the mixing of wastewater with facility NCCW and the subsequent discharge to the Mississippi River. According to 3M Cordova records, the average daily flow from outfall A01 is 0.7 MGD, and the average daily flow from outfall 001 is 7.8 MGD.

Analyte	**Sample VP1364-15** Outfall A01 (Pond #3) (sample collected from discharge stream directly downstream of the V-notch weir) **Analyte Concentration (ng/L)**	**Sample VP1364-16** Lift station B63 (sample collected from wet well in building of lift station B63) **Analyte Concentration (ng/L)**	**Sample VP1364-17** Building 30 and thermal oxidizer discharge (sample collected from discharge pipe from building 30 and thermal oxidizer in junction box) **Analyte Concentration (ng/L)**	**Sample VP1364-18** Building 20 discharge (sample collected from discharge pipe from building 20 in junction box) **Analyte Concentration (ng/L)**
PFBA	233,000	351,000	1,090,000	41,400
PFDA	125	151	152	175
PFDoA	10.6	Not observed	Not observed	Not observed
PFHpA	407	330	445	242
PFHxA	1,650	934	1,280	964
PFNA	599	864	577	> 515[b]
PFOA	778	718	587	829
PFPeA	9,390	8,460	10,500	7,220
GenX	7,110	3,040	4,660	777
PFECA-A	2,460	2,220	9,791	219
PFECA-B	Not observed	9.12	36.0	1.31
PFECA-G	Not observed	21.4	69.2	5.75
FOSA	11.8	13.7	5.48	13.0
N-MeFOSE	Not observed	Not observed	Not observed	40.8
PFBS	38,000	109,000	22,600	55,500
PFHpS	316	101	356	87.8
$PFHxS_{Total}$	2,140	1,380	1,210	2,260
PFNS	19.5	Not observed	Not observed	Not observed
$PFOS_{Total}$	11,600	> 4,630[b]	9,470	> 417[b]

Observation: CWA 11

PFPeS	346	355	563	75.3
6:2 FTS	155	1.71	Not observed	1.98
8:2 FTS	Not observed	Not observed	2.61	Not observed
10:2FTS	4.02	Not observed	0.816	Not observed

In addition to the quantitative analysis, NEIC performed a qualitative analysis of the samples for the presence of 34 PFAS. The following PFAS were observed in samples VP1364-15, VP1364-16, VP1364-17, and VP1364-18.

PFAS observed in sample VP1364-15

- 4,4'-(Hexafluoro-isopropylidene)diphenol
- FBSAA(1) (Perfluorobutyl sulfonamido acetic acid)
- Gen-X (Undecafluoro-2-methyl-3-oxahexanoic acid)
- HQ-115 (Bis(trifluoromethylsulfonyl)amine)
- PBSA (1-Butanesulfonamide, N-(3-(dimethylamino)propyl)-1,1,2,2,3,3,4,4,4-nonafluoro-)
- PBSA-C1 (3-{[3-Dimethylamino)propyl](nonafluorobutane-1-sulfonyl)amino}propanoic acid)
- PFBSi (Perfluorobutanesulfinic Acid)
- PFEESA (Perfluoro(2-ethoxyethane)sulfonate)
- PFESA Byproduct 1 (Perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid)

PFAS observed in sample VP1364-16

- FBSA (Perfluorobutane sulfonamide (C4 amide) 1,1,2,2,3,3,4,4,4-Nonafluorobutane-1-sulfonamide)
- Gen-X (Undecafluoro-2-methyl-3-oxahexanoic acid)
- HQ-115 (Bis(trifluoromethylsulfonyl)amine)
- PBSA (1-Butanesulfonamide, N-(3-(dimethylamino)propyl)-1,1,2,2,3,3,4,4,4-nonafluoro-)
- PFBSi (Perfluorobutanesulfinic Acid)
- PFEESA (Perfluoro(2-ethoxyethane)sulfonate)
- PFES (Perfluoroethanesulfonate)
- PFESA Byproduct 1 (Perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid)

PFAS observed in sample VP1364-17

- FBSA (Perfluorobutane sulfonamide (C4 amide) 1,1,2,2,3,3,4,4,4-Nonafluorobutane-1-sulfonamide)
- Gen-X (Undecafluoro-2-methyl-3-oxahexanoic acid)
- PFBSi (Perfluorobutanesulfinic Acid)
- PFEESA (Perfluoro(2-ethoxyethane)sulfonate)
- PFES (Perfluoroethanesulfonate)
- PFPS (Perfluoropropanesulfonate)

PFAS observed in sample VP1364-18

- FBSA (Perfluorobutane sulfonamide (C4 amide) 1,1,2,2,3,3,4,4,4-Nonafluorobutane-1-sulfonamide)
- Gen-X (Undecafluoro-2-methyl-3-oxahexanoic acid)
- HQ-115 (Bis(trifluoromethylsulfonyl)amine)
- PFBSi (Perfluorobutanesulfinic Acid)
- PFECA F or PMPA (2,2,3,3-tetrafluoro-3-(trifluoromethoxy)-propanoic acid)
- PFEESA (Perfluoro(2-ethoxyethane)sulfonate)
- PFES (Perfluoroethanesulfonate)
- PFESA Byproduct 1 (Perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid)
- PFPS ((Perfluoropropanesulfonate)

Observation: **RCRA 1**

Observation Summary: Nine containers of hazardous waste were not labeled as "Hazardous Waste."

Observation: **RCRA 1**

Citation:

35 Illinois Administrative Code (IAC) § 722.117(a)(5)(A)(i) [40 CFR § 262.17(a)(5)(i)(A)] – *a)* *Accumulation. The LQG may accumulate hazardous waste on site for no more than 90 days, unless in compliance with the accumulation time limit extension or F006 accumulation conditions for exemption in subsections (b) through (e). The following accumulation conditions also apply:*
5) Labeling and Marking of Containers and Tanks
A) Containers. An LQG must mark or label its containers with the following:
i) The words "Hazardous Waste"

Evidence:

Appendix RCRA C – NEIC RCRA Photographs

Description of Observation: On December 6, 2019, nine containers of hazardous waste were not labeled as "Hazardous Waste":

- Two trailers at loading station 83 (**Appendix RCRA C**, photographs 31-34)
- Seven 55-gallon drums located in the building 2 accumulation area. These drums are 55-gallon steel drums, painted yellow on top and green on the bottom. One drum was placed inside a pink plastic bag because, according to facility personnel, it was leaking (**Appendix RCRA C**, photographs 21, 23, and 24).

Observation: **RCRA 2**

Observation Summary: Twelve containers of hazardous waste were not dated with an accumulation start date.

Citation:

35 IAC § 722.117(a)(5)(A)(iii) [40 CFR § 262.17(a)(5)(i)(C)] – *a) Accumulation. The LQG may accumulate hazardous waste on site for no more than 90 days, unless in compliance with the accumulation time limit extension or F006 accumulation conditions for exemption in subsections (b) through (e). The following accumulation conditions also apply:*
5) Labeling and Marking of Containers and Tanks
A) Containers. An LQG must mark or label its containers with the following:
iii) The date upon which each period of accumulation begins clearly visible for inspection on each container.

Evidence:

Appendix RCRA C – NEIC RCRA Photographs

Description of Observation: On December 6, 2019, 12 containers of hazardous waste were not marked with the date upon which each period of accumulation begins:

- Two trailers at loading station 83 (**Appendix RCRA C**, photographs 31-34)
- One trailer located at building 18 (**Appendix RCRA C**, photographs 10, 11, and 12**)**
- Seven 55-gallon drums located in the building 2 accumulation area. These drums are 55-gallon steel drums, painted yellow on top and green on the bottom. One drum was placed inside a pink plastic bag because, according to facility personnel, it was leaking (**Appendix RCRA** C, photographs 21, 23, and 24)**.**
- Two additional 55-gallon drums located in the building 2 accumulation area (**Appendix RCRA C**, photographs 25 and 26).

Observation: **RCRA 3**
Observation Summary: Nine containers of hazardous waste were stored in a manner that could rupture the container and were not stored in a manner that minimizes the possibility of a release of hazardous waste.
Citation: **35 IAC § 722.117(a)(1)(D)(ii) [40 CFR § 262.17(a)(1)((iv)(B)** – *D) Management of Containers ii) The LQG must not open, handle, or store a container holding hazardous waste in a manner that may rupture the container or cause the container to leak.* **35 IAC § 722.351 [40 CFR § 262.251]** – *An LQG must maintain and operate its facility in a manner that minimizes the possibility of a fire, explosion, or any unplanned sudden or non-sudden release of hazardous waste or hazardous waste constituents to air, soil, or surface water which could threaten human health or the environment.*
Evidence: **Appendix RCRA C** – NEIC RCRA Photographs
Description of Observation: On December 6, 2019, nine containers of hazardous waste were found collapsed: two containers located at building 18 (**Appendix RCRA C**, photographs 8 and 9) and seven containers at the silo 90-day hazardous waste accumulation area **Appendix RCRA C**, photographs 27-30). Discussions with facility personnel indicated that the waste stream is hot when generated, and as the waste cools, the sides of the drums collapse. The hot waste is collected in black polyethylene drums, not steel, because of other characteristics of the waste. Deformed drums were located on the bottom of drum stacks that were at least two sets of pallets high.

Observation: **RCRA 4**
Observation Summary: Two 90-day areas were being operated without adequate aisle space between containers of hazardous waste.
Citation: **35 IAC 722.355 (40 CFR § 262.255)** – *The LQG must maintain aisle space to allow the unobstructed movement of personnel, fire protection equipment, spill control equipment, and decontamination equipment to any area of facility operation in an emergency, unless aisle space is not needed for any of these purposes.*
Evidence: **Appendix RCRA C** – NEIC RCRA Photographs
Description of Observation: On December 6, 2019, two 90-day areas were being operated without adequate aisle space between containers of hazardous waste. At the building 18 90-day area, metal totes were stacked so tightly together that there was no access to the containers stacked in the middle of the area (**Appendix RCRA C**, photographs 6 and 7). Containers in the building 2 90-day area were stacked in a U-shape; containers in the corner of the "U" were inaccessible.

Observation: **RCRA 5**
Observation Summary: Six containers labeled with "Waiting Analysis" labels were stored beyond the 90-day accumulation limit.
Citation: **35 IAC § 722.117 (40 CFR § 262.17)** – *An LQG may accumulate hazardous waste on site without a permit or interim status, and without complying with the requirements of 35 Ill.*

Observation: **RCRA 5**
Adm. Code 702, 703 and 724 through 727 and the notification requirements of section 3010 of RCRA (42 USC 6930), provided that the LQG meets all of the following conditions for exemption: *a) Accumulation. The LQG may accumulate hazardous waste on site for no more than 90 days, unless in compliance with the accumulation time limit extension or F006 accumulation conditions for exemption in subsections (b) through (e).*
Evidence: **Appendix RCRA C** – NEIC RCRA Photographs
Description of Observation: On December 6, 2019, six containers of hazardous waste located in the building 2 90-day area were stored beyond the 90-day accumulation limit. The containers were labeled with a "Waiting Analysis" label, which also contains the words "Hazardous Waste" (**Appendix RCRA C**, photographs 14, 15, 17, 18, 20, and 22). Two of the containers were not dated with an accumulation start date (**Appendix RCRA C**, photographs 20 and 22), and the facility provided no information as to when the containers were put into the 90-day area. The other four containers were dated: August 2018 (**Appendix RCRA C**, photograph 18), July 2019 (**Appendix RCRA C**, photograph 14), and August 2019 (two containers) (**Appendix RCRA C**, photographs 15 and 17).